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EFFECT OF REACTOR IRRADIATION ON STRESS-RUPTURE STRENGTH OF AUSTENITIC STEELS AND HEAT-RESISTANT MATERIALS BASED ON IRON AND NICKEL

by E.V.Gusev, P.A.Platonov,
N.F.Pravdyuk, N.M.Sklyarov

INTRODUCTION

Until recently it was believed that irradiation of structural materials at high temperature should not cause considerable changes of their properties, because it was observed that in most cases irradiated materials restore their original properties when annealed or tested at elevated temperatures. However it can be seen from the description of some experiments that follows that a number of materials when irradiated in the high temperature range exhibited most drastic changes, which is an indication of appearance in a material structure of the changes that are difficult to remove.

TEST MATERIALS AND TYPES OF TESTS

Investigation was carried out with six alloys which were so selected that their composition and structure corresponded to the specific features of heat-resistant and stainless steels and alloys. In addition, commercial nickel (99.95%) was also tested. The chemical composition [1] of alloys is presented in Table 1. Amongst the materials so far tested were both age-hardenable alloys (XH77TЮP, X12H22T3MP, alloy I, steel II) and alloys similar to homogenious solid solutions (IXI8H9T , XH60B) .

All the alloys, excluding steel type IXI8H9T, were tested after usual heat treatment. Steel type IXI8H9T and nickel were tested in the delivery stage.

The irradiation of specimens was carried out in the fuel channels of the reactor RFT with the maximum thermal neutron flux of 8×10^{13} n/cm^2.sec ; the fast neutron flux being 5×10^{13} n/cm^2.sec ($>$ 1 Mev). Hereafter integral

radiation fluxes will be indicated only for fast neutrons . The usual irradiation technique was used, which was described earlier [2,3]. Studies were made of mechanical properties during tensile tests , as well as of hardness, creep strength and microstructure.

EFFECT OF IRRADIATION ON TENSILE STRENGTH

Investigation into the effect of irradiation at 150-200°C on the tensile strength of heat-resistant steels and alloys has shown that their mechanical properties, as measured at room temperature after receiving a comparatively high integrated irradiation dose (up to 10^{20} n/sq.cm), vary within the following limits. The yield stress rose by 10-30 per cent, relative elongation decreased by 30-40 per cent. Somewhat unexpected was the decrease in the ultimate tensile strength of the alloys XH77TЮP, XI2H22T3MP and XH60B after irradiation.

Such changes in heat-resistant alloys, which are characterized by a large plasticity margin in the initial state, cause no serious fear when they are used at room temperature. At elevated temperatures, however, quite substantial changes in their mechanical properties are revealed.

Figs.1 and 2 depict the results of mechanical tests of irradiated and unirradiated specimens of the alloys XH77TЮP and XH60B as a function of the test temperature. The irradiation was carried out at 150-200°C, the integrated dose being approximately $(1-3)\times10^{20}$ n/sq.cm. It can be seen that beginning with 500-550°C the mechanical properties of irradiated materials undergo drastic changes. Particularly conspicuous is the change in relative elongation which reduced practically to zero in the alloy XH77TЮP and to 5-7 per cent in the alloy XH60B.

Tests of irradiated specimens at elevated temperatures were run with other alloys, too. Thus, for steel X12H22T3MP we also studied the effect of irradiation on mechanical characteristics at temperatures up to 750°C (i.e. in the temperature range where alloys of this type are used). The results of tensile tests of irradiated and unirradiated specimens of steel X12H22T3MP at 20 and 750°C have shown that at 750°C plasticity drops abruptly from 16 to 3 per cent.

The ultimate strength and the yield stress of irradiated specimens decreased much less than those of unirradiated specimens (by 3-5 and 7-10 per cent, respectively).

Comparing the results of the tests of the investigated materials, XH77TЮP, X12H22T3MP and XH60B one can see that at high temperatures the nature of the changes in the properties due to irradiation is nearly identical for all the alloys. Somewhat sharper changes were observed in the alloys XH77TЮP and X12H22T3MP. The alloy XH77TЮP, for instance, does not show any noticeable deformation at the test temperature of 750°C, whereas in the case of the alloy XH60B. deformation reaches about 7 per cent.

Very interesting results were obtained also during the tests of commercial nickel irradiated with a fast neutron integrated flux of 1.7×10^{20} n/sq.cm at 150-200°C.

Fig.3 displays the results of tensile tests of nickel before and after irradiation as a function of the test temperature.

The graph of Fig.3 shows that in the case of irradiated nickel, as well as alloys based on it (see Figs.1 and 2) a drastic decrease in plasticity is observed in the vicinity of 600°C. The difference in the plasticity of irradiated and unirradiated nickel at temperatures above 600°C is particularly conspicucus because the nickel was investigated in a deformed state. It can be seen that, beginning with 600°C, the plasticity of unirradiated nickel increases considerably, which fact is attributed to the restoration of the original properties of the deformed material, whereas irradiated nickel exhibits an abrupt drop in plasticity in this temperature range.

It should be noted that a deterioration in properties of alloys after irradiation is revealed not only in tests at high temperatures, but also at room temperature after heating by 700-800°C. Thus, the results of a room temperature tensile test of irradiated specimens of the alloy XH77TЮP before and after 1 hr exposure to a temperature of 750°C in a vacuum have shown that in the case of the irradiated alloy, only the yield stress is restored. Ultimate tensile strength and especially relative elongation decrease (approximately by 10 and 30 per cent, respectively).

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Apart from tensile tests at high temperatures, hot hardness measurements were made. In Fig.4 we present the results of the measurements of hot hardness of the alloy XH77TЮР and nickel as a function of the test temperature. The alloy XH77TЮР was tested for hardness in two states: quenched and then aged, and just quenched. The hardness of irradiated specimens of the investigated materials at room temperature is considerably higher than for unirradiated ones. As the test temperature increases, the hardness of irradiated materials approximates that of unirradiated ones, and at 600°C their values become practically identical. Thus, if during tensile tests the difference in the properties of irradiated and unirradiated materials at high temperatures sharply increases, hardness measurements at these temperatures yield almost identical hardness values. This discrepancy indicates that the behaviour of irradiated heat-resistant materials depends on the stressed state during deformation, and in this case no correlation is observed between the hot hardness and the strength of the materials. This may be associated with the fact that the rupture strength of the material changes irreversibly while shear strength restores on heating.

Thus, tensile tests of the investigated alloys at elevated temperatures demonstrate that irradiation causes considerable changes in alloys, which are hardly revealed during tests at room temperature. For these alloys, the changes are characterized by the fact that in a certain temperature range (550–800°C) there is an abrupt drop in plasticity which in this case appears to be a characteristic most sensitive to irradiation. For some alloys, the drop in plasticity is accompanied also by a substantial decrease in ultimate strength.

EFFECT OF IRRADIATION ON STRESS-RUPTURE STRENGTH

Stress-rupture strength tests were run with all the materials considered in the preceding section. Besides, such materials as IXI8H9T, the alloy I (with and without boron) and steel II were tested.

Comparative tests of irradiated and unirradiated specimens of the investigated materials in the temperature range

from 600 to 900°C have demonstrated a considerable decrease in stress-rupture strength. For some nickel-and iron-base alloys, the decrease proved to be absolutely catastrophic.

The lowest radiation resistance was revealed in the alloy XH77TЮР. Irradiated specimens of the alloy XH77TЮР proved to be so unstable under constant load at 750°C that even at a stress of 8 kg/sq.mm the failure time was 0.5 hr at the most, while unirradiated specimens failed only after 30-50 hrs even at a stress of 35 kg/mm^2. At such stresses irradiated specimens failed instantly. It was only at a stress of 5 kg/sq. mm (55-70 hrs) that the failure time for irradiated specimens could be compared with the failure time of unirradiated specimens at a stress of 35 kg/sq.mm.

Thus, irradiation with an integral flux of $(1-3)\times10^{20}$ n/cm^2 leads to a more than six-fold reduction in the rupture stress for the alloy XH77TЮР at a test temperature of 750°C, i.e. after irradiation this high-strength nickel alloy becomes less heat-resistant than many of the conventional low-alloy steels.

As can be seen from Fig.9, a decrease in the stress-rupture strength of the alloy XH77TЮР after irradiation takes place also at other temperatures (600 and 800°C). The decrease in stress-rupture strength is so great that after irradiation the alloy XH77TЮР became much less heat-resistant at 600°C than its unirradiated specimen was at 800°C.

An increase in the irradiation temperature to 700°C practically does not decrease the irradiation effect.

Unirradiated steel X12H22T3MP is approximately equivalent in heat-resistance to the alloy XH77TЮР. The failure time for this steel at 750°C and a stress of 35 kg/mm^2 is about 25-50 hrs. At this stress, irradiated specimens fail at the moment of loading. After irradiation this steel exhibited a somewhat higher resistance at a stress of 17.5 and 8 kg/mm^2 than the alloy XH77TЮР (3 hours and 30-80 hours respectively).

The alloy XH60B is less sensitive to neutron irradiation; at a temperature of 800°C and the failure time of 50 hours the stress-rupture is decreased by 20-30%.

A specimen of the alloy I was melted for investigating the role of boron in high-temperature embrittlement of heat-resistant alloys. Alloy ingots were prepared in two pourings;

in the first case boron was added in the amount of 0.015 wt.%, while in the second, only boron traces were present (<0.001 wt.%). To exclude the effect of secondary constituents, the alloy was prepared from as pure burden materials as possible.

The test results suggest that boron addition does not play a decisive role in the changes of stress-rupture strength after irradiation. Practically similar results were obtained also during tests of specimens of high-alloy foundry steels where the boron content was also 0.001 per cent in the first and 0.015 per cent in the second pouring.

Effects detected in heat-resistant austenitic steels suggested that a decrease in stress-rupture strength as a result of irradiation is possible also in stainless steels grade 18-8. The tests carried out confirmed this supposition.

Fig.6 demonstrates the results of tests for stress-rupture strength of stainless steel IXI8H9T before and after irradiation at temperatures of 450 and 550°C with an integrated flux of $(1\text{-}3) \times 10^{20}$ n/sq.cm.

It can be seen from the figure that although irradiation leads to a marked decrease in stress-rupture strength, it is not so great as for heat-resistant materials. Thus, the stress-rupture strength of steel IXI8H9T after irradiation at a test temperature of 700°C and a failure time of 50 hrs decreased by about 15 per cent. It should be noted that, as in the case of the alloy XH77TЮP, changes in the stress-rupture strength of stainless steel are practically independent of the irradiation temperature.

In addition to stainless steel IXI8H9T, tests were made with ferro-nickel-chromium steel II alloyed with tungsten and hardened by precipitation hardening.

Fig. 7 depicts the results of testing this steel for stress-rupture strength at test temperatures of 600,700 and 800°C before and after irradiation at temperatures of 450 and with an integral flux of $(1\text{-}3) \times 10^{20}$ n/sq.cm. It can be seen that for this steel, changes in stress-rupture strength are considerably greater than for steel 18-8 and comparable with changes observed for heat-resistant alloys.

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Tensile tests of nickel, during which a very drastic change in plasticity was revealed in the temperature range above 500–600°C, gave grounds to expect a marked decrease in stress-rupture strength too. Although stress-rupture strength drops noticeably, its decrease is less than for all the investigated materials. This difference between nickel and the other tested materials may evidently be explained by the fact that although the plasticity of nickel in the region of 600°C falls off sharply, its ultimate strength remains at the same level (or even slightly higher) than for unirradiated nickel (see Figs.6 and 7).

In order to check the possibility of restoring the properties of the alloys XH77TЮP and XI2H22T3MP, irradiated specimens were exposed to a temperature of 750°C for 10 hrs. This temperature is somewhat higher than the temperature of annealing of radiation defects in nickel and iron and corresponds to the temperature at which work hardening by precipitation hardening is possible. Such an exposure, however, does not result in a niticeable restoration of the heat-resistance of materials, as can be seen from Table II.

An attempt was made to restore the properties of the alloys XH77TЮP and XI2H22T3MP by a complete heat treatment cycle for irradiated specimens. Additional heat treatment in a vacuum was carried out according to the following schedule: (a) for the alloy XH77TЮP : homogenization at 1,080°C for 1 hr followed by aging at 750°C for 16 hrs; (b) for the alloy XI2H22T3MP: homogenization at 1,150°C for 30 min., exposure at 750°C for 16 hrs, then decreasing the temperature to 650°C with a 16-hr exposure. It appears, however, that even additional heat treatment does not restore the characteristics of the instantaneous and stress-rupture strength of these alloys.

METALLOGRAPHIC INVESTIGATION

Selective metallographic investigations have been made with a view to evaluating the effect of irradiation on the structural characteristics of heat-resistant steels and alloys. The microstructure of the heads of irradiated and unirradiated tensile specimens of the investigated materials did not differ noticeably. It was only noted that the grain boundaries of irradiated specimens were etched more strongly than in unirradiated ones.

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During metallographic studies of the working part of specimens tested for instantaneous and stress-rupture strength in the temperature range from 600 to 800°C, a substantial difference in the deformation of grains in irradiated and unirradiated specimens was noted. The grains of irradiated specimens are much less deformed in the direction of the load applied, as compared with unirradiated specimens. The difference in the nature of grain deformation increases with test temperature.

Metallographic investigation of specimens subjected to tests for stress-rupture strength showed that brittle fracture occurs at the grain boundary without any noticeable deformation of the grain. The microstructure of a specimen of the alloy ХН77ТЮР tested for stress-rupture strength is presented in Fig. 8. The section was prepared from the non-fractured part specimen which was under the same stress as the fractured portion.

DISCUSSION OF RESULTS

It has been established that reactor irradiation causes substantial in the mechanical properties of heat-resistant alloys based on iron and nickel and of austenitic chromium-nickel steels, these changes occurring in the high-temperature range. The changes are characterized by the fact that at high tests temperatures, beginning with 500-600°C, the plasticity and strength of the alloys decrease considerably. In most cases, the changes are revealed already in short-term tests at high temperatures.

In long-term tests a decrease in stress-rupture strength was revealed for all the investigated alloys. For the majority of heat-resistant alloys the changes in stress-rupture strength are much greater than those observed as a result of heat treatment, pressure-working and other technological processes.

When comparing the investigation results for various alloys, it can be seen that alloys with a more non-uniform structure show greater changes in stress-rupture strength. For instance, changes in stress-rupture strength are much greater

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for the alloys XH77TЮP and XI2H22T3MP which have the hardening phases in their structure, than for the alloy XH60B which represents an almost uniform solid solution hardened only with a small amount of cubic carbides. A similar conclusion concerning the role of disperse phases in radiation brittleness may be made from a comparision of the results of tests of steel IXI8H9T and steel II. However, the considerable changes in the properties revealed during nickel tests suggest that structure uniformity is only one of the many factors affecting the high-temperature brittleness of heat-resistant materials.

A characteristic feature of changes occurring on irradiation is their irreversibility. Properties are restored neither on prolonged heating in the range of temperatures corresponding to the annealing of radiation defects in nickel and iron, nor on additional heat treatment.

Such peculiarities in the behaviour of irradiated materials at high temperatures indicate that high-temperature brittleness of heat-resistant alloys and austenitic steels is due to complex processes which include both the effects of radiation damage and the effects caused by nuclear transformations and, possibly, some physico-chemical processes.

It could be assumed that the drastic change in properties is associated with the possibility of phase transformations due to irradiation. Since the first data on the changes in stress-rupture strength were obtained during the study of complex-composition alloys (XH77TЮP and others), such an assumption could be justified. It could, however, be supposed that additional heat treatment should bring the alloy back to the initial state and eliminate all the phase transformations which might have occurred in the course of irradiation; nevertheless, complete heat treatment of alloys which included homogenization, hardening and aging did not result in a noticebly restoration of the properties. Besides, tests of nickel specimens prove convincingly enough that phase transformations could not be the principal cause of the drastic deterioration in the properties of irradiated materials at high temperatures.

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It seems most probable that the cause of such irreversible changes lies in radiation alloying due to the presence of new impurities appearing as a result of nuclear reactions.

Reactions of the type (n, γ) cannot give rise to serious changes since they lead to the formation of atoms whose properties differ little from the original ones, and their amounts are toc small to cause noticeable changes. Therefore the most probable cause of changes in properties at high temperatures is the development of such nuclear reactions which lead to the formation of elements widely differing from the initial ones in their properties.

The most dangerous, from the point of view of a possible deteroration of properties, are those reactions which may result in the formation of a gaseous phase in metals. The most probable sources of the formation of a gaseous phase appear to be the reaction $B^{10} (n, \alpha) Li^7$ and $Ni^{58} (n, p) Co^{58}$.

Though the latter reaction produces comparatively small amount of hydrogen but the fact that so far high-temperature embrittlement was observed only for nickel-containing alloys shows that this reaction cannot be neglected.

As the volume of gas-filled cavities is determined by the energy of free surface formation and since the surface energy has the minimum value at the grain boundaries, the formation of the largest gas volumes may be expected just at the grain boundaries or at the phase interfaces. Owing to this the grain boundaries may be greatly weakened, and this is most pronounced at the temperature range above equicohesive one, where cohesion at the grain boundaries is below grain strength. The irradiation leads apparently not only to the formation of gaseous products as a result of nuclear reactions but it should also substantionally change the conditions for the formation of gas volumes, due to the formation of zones with an increased concentration of vacancies that may act as nucleies of pores. The major part of the mentioned data is in agreement with the supposition that the formation of gas volumes at the grain boundaries is the most probable cause of high temperature embrittlement of alloys.

In conclusion, the authors deem it their duty to thank A.D.Amayev who helped them to carry out the tests, as well as V.G.Dorofeyev, V.A.Nikolaev and A.N.Lapin who took part

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in investigating stainless steels.

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Table 1

No.	Steel or alloy grade	Content of elements, % (nominal)										
		C	Si	Mn	Cr	Ni	Ti	Al	Mo	Fe	B	Miscellaneous elements
1	XH77TЮP	≤ 0.06	≤ 0.6	≤ 0.40	19.0–22.0	base	2.30–2.70	0.55–0.95	–	≤ 4.0	≤ 0.01	Ze(< 0.01)
2	XI2H22T3MP	≤ 0.10	≤ 0.60	≤ 0.60	10.0 12.5	21.0–25.0	2.60–3.20	≤ 0.80	1.00–1.60	base	0.02	
3	XH60B	≤ 0.10	≤ 0.80	≤ 0.50	23.5–26.5	base	0.30–0.70	≤ 0.50	–	≤ 4.0	–	W(13.0–16.0)
4	Alloy I x)	–	–	–	–	base	2.3	6.0	17	–	traces	
5	IXI8H9T	≤ 0.12	≤ 0.80	1.00–2.00	17.0–19.0	9.0–11.0	0.50–0.70	–	–	base	–	
6	Steel II	≤ 0.10	≤ 0.70	1.00–1.60	17.0–20.0	21.0–24.0	1.0–1.5	–	–	base	–	W

x) The alloy I was melted without boron (first pouring) and with boron (second pouring). In the ingots of the second pouring the boron content did not exceed 0.015 per cent.

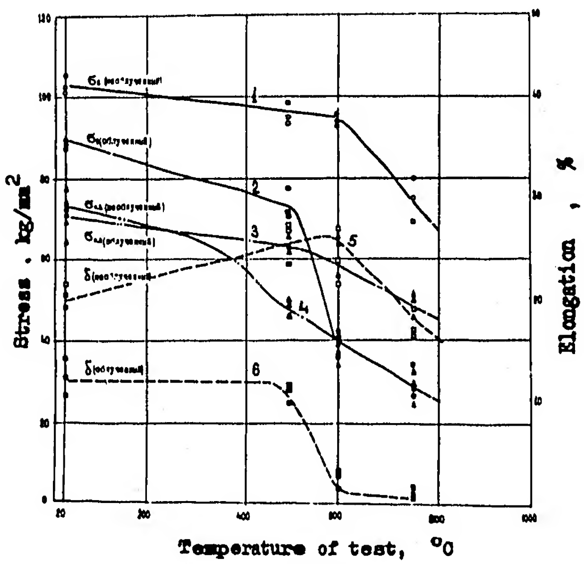

Fig.1. Mechanical properties of the alloy XH77TЮP as a function of test temperature
1,3 and 5 - unirradiated specimens; 2,4 and 6 - specimens irradiated at 150-200°C with an integrated flux of $(1\text{-}3)\times10^{20}$ n/cm^2

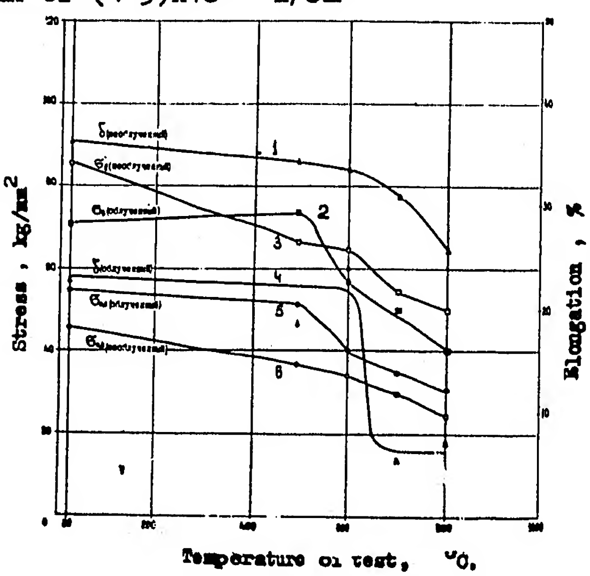

Fig.2. Mechanical properties of the alloy XH60B as a function of tests temperature
1,3,6 - unirradiated specimens; 2,4 and 5 - specimens irradiated at 150-200°C with an integrated flux of $(1\text{-}3)\times10^{20}$ n/cm^2

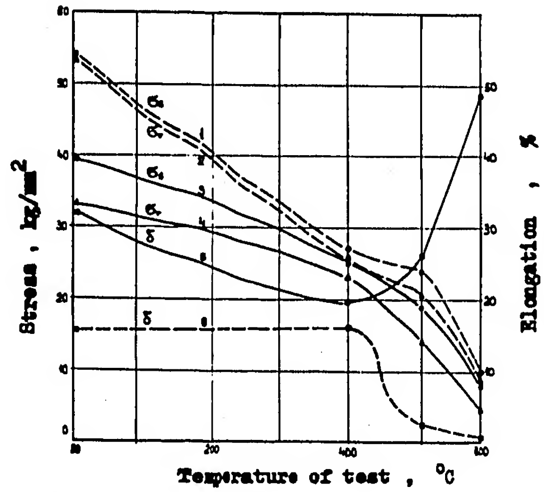

Fig.3. Mechanical properties of commercial nickel as a function of test temperature

1,2 and 6 – unirradiated specimens; 3,4 and 5 - specimens irradiated at 150-200°C with an integrated flux of 1.7 x 10^{20} n/cm^2

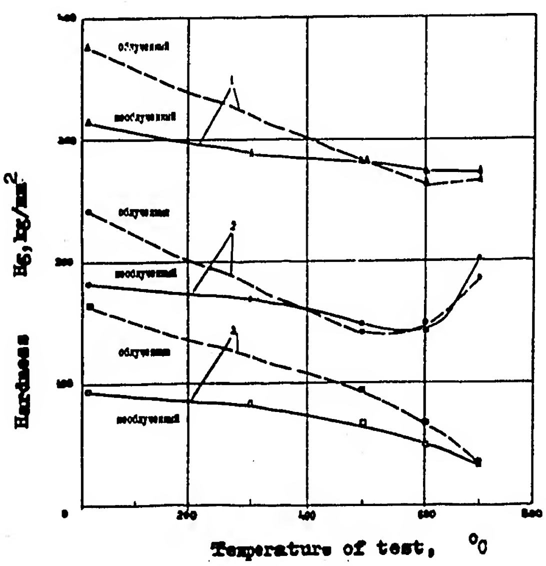

Fig.4. Hardness of the alloy XH77TЮP and commercial nickel as a function of test temperature

1 – alloy XH77TЮ P in a quenched and aged condition;

2 – alloy XH77TЮ P in a quenched condition;

3 – nickel

Continuous lines – unirradiated specimens,

dotted lines – specimens irradiated

at 150-200°C with an integrated flux of 1.3x10^{20} n/cm^2

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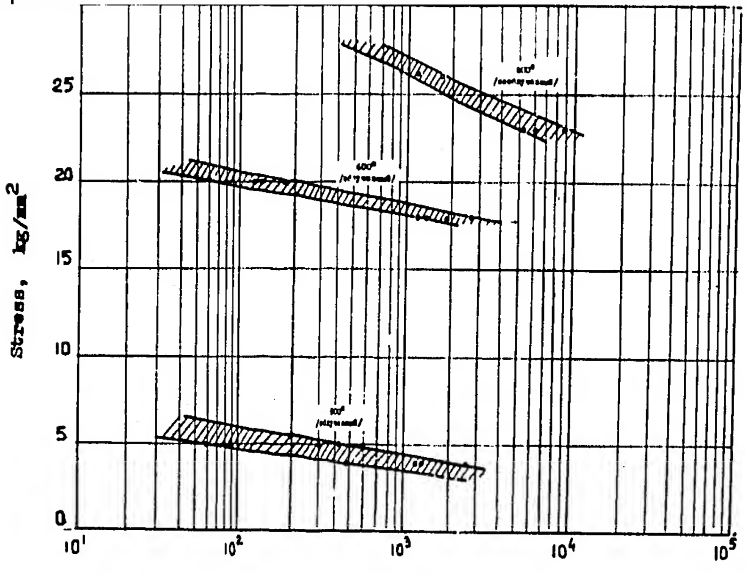

Fig.5. Dependence of failure time on applied stress of the alloy XH77TЮP at 600 and 800°C. 0 - unirradiated specimens; •-specimens irradiated at 150-200°C with an integrated flux of $(1-3)\times10^{20}$ n/cm^2

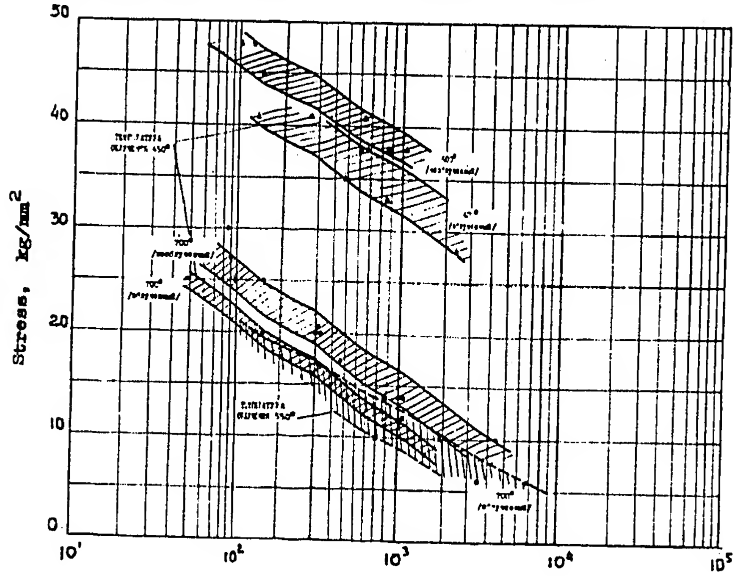

Fig.6. Dependence of failure time on applied stress of steel IXI8H9T at 600 and 700°C

○△ - unirradiated specimens;

●▲x-specimens irradiated at 450 and 550°C with an integrated flux of $(1-3)\times10^{20}$ n/cm^2

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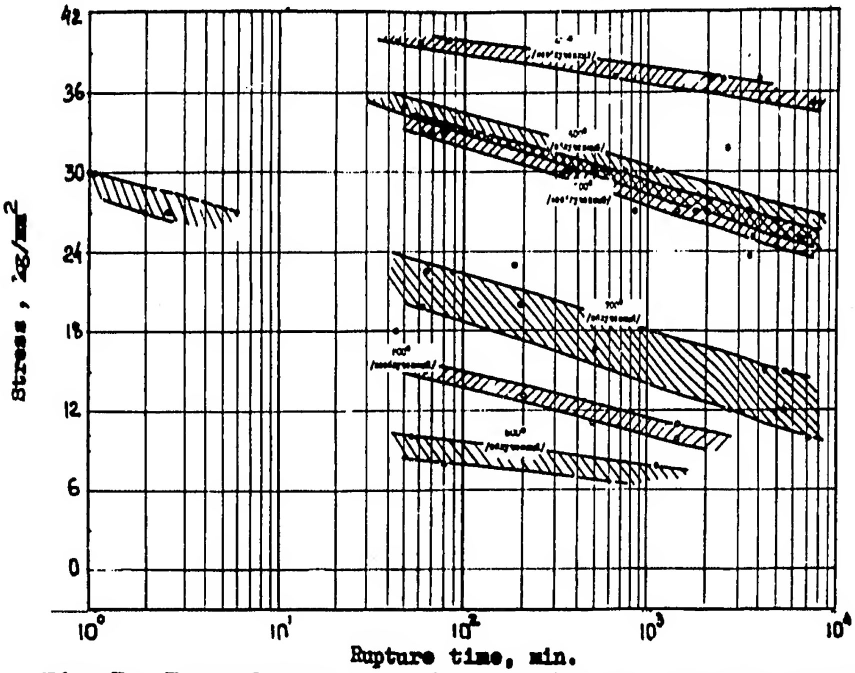

Fig.7. Dependence of failure time on applied stress of steel II at 600, 700 and 800°C.

0 - unirradiated specimens;

● - specimens irradiated at 450°C with an integrated flux of $(1-3)\times 10^{20}$ n/cm^2

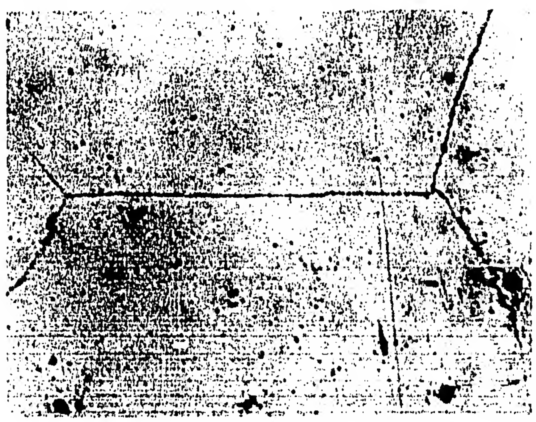

Fig.8. Appearance of cracks arising in an unfailed necked part of a specimen of the alloy XH77ТЮР irradiated at 150-200°C with an integrated flux of $(1-3)\times10^{20}$ n/cm^2 after a creep rupture test at a temperature of 800°C and a stress of 5 kg/mm^2. The section was not etched (600 X).

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